

Local density of states study of a spin-orbit-coupling induced Mott insulator Sr_2IrO_4

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We present scanning tunneling microscopy and spectroscopy experiments on the novel $J_{\text{eff}} = 1/2$ Mott insulator Sr_2IrO_4 . Local density of states (LDOS) measurements show an intrinsic insulating gap of 620 meV that is asymmetric about E_F and is larger than previously reported values. The size of this gap strongly suggests that Sr_2IrO_4 is a Mott rather than Slater insulator. In addition, we found a small number of native defects which create in-gap spectral weight. Atomically resolved LDOS measurements on and off the defects naturally explain why such a large gap in this 5d system has not been seen by other probes. Together the extended nature of the 5d electrons and poor screening of defects help explain the illusive nature of this gap.

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Unlike localized 3d electrons and their resultant Mott insulating states [1], 4d and 5d electrons are more extended in space and thus should have both reduced on-site Coulomb repulsion (U) and enhanced energy bandwidths (W). Because of this, many 4d systems [2–4] are close to the borderline ($U \sim W$) of localization and one might expect that 5d systems will be metallic ($U < W$). However, many iridium oxides [5–8] have instead been found to be insulators. Recently, it has been shown that for octahedrally coordinated Ir^{4+} ($5d^5$) the large spin-orbit coupling (SOC), inherent to such a heavy element ($Z = 77$), can dominate the t_{2g} orbitals and split them into a $J_{\text{eff}} = 1/2$ doublet and a $J_{\text{eff}} = 3/2$ quartet [9]. In this strong SOC limit, the quartet band is fully occupied and the conduction band becomes the half-filled $J_{\text{eff}} = 1/2$ band with a much smaller W . This means that the iridium oxides can be driven across the Mott transition borderline with a much smaller U .

One of these 5d materials, Sr_2IrO_4 , is of particular interest, since it is the first material shown to have this SOC-induced insulating state [9, 10]. In addition, it bears many similarities with the well-studied La_2CuO_4 [11], a parent compound of the high- T_c superconductors, leading to an appealing possibility of superconductivity in Sr_2IrO_4 with hole doping [12, 13]. However, one of the similarities to La_2CuO_4 , which relies on Sr_2IrO_4 being in the strong SOC limit so that only the $J_{\text{eff}} = 1/2$ band needs to be considered, becomes less certain when one realizes that the electron hopping energy ($t \approx 0.3$ eV [12, 14]) is close to the SOC energy ($\zeta \approx 0.5$ eV [15]). The deviation from the strong SOC limit has been confirmed by the recent work by Haskel *et al.* [16]. Furthermore, instead of the Mott type insulator, the magnetically originated Slater insulator has also been proposed [17], partially supported by time-resolved optical measurements [18].

A key to identify the nature of this insulator is to answer the important questions of what is the size of the insulating gap, Δ , and how does it compare to other en-

ergy scales (e.g. t and ζ). Surprisingly, measurements of the gap size from different probes in Sr_2IrO_4 vary widely. Gaps as low as ~ 0.1 eV have been reported by fitting the resistivity data with a thermal activation model [19, 20] and by calculations using local density approximation (LDA) with SOC and U [9, 21]. On the other hand, angle-resolved photoemission spectroscopy (ARPES) measurements do not agree with this value since the maximum of the valance band is already lower than -0.1 eV [9, 22]. Optical conductivity and resonant inelastic x-ray scattering (RIXS) studies both yield $\Delta \leq 0.4$ eV [9, 11, 23]. Importantly, these measurements are either indirect, susceptible to signals from other excitations like excitons or magnons, or can only measure the filled states, making the interpretations of their gap values difficult. To resolve these discrepancies, it is highly desirable to measure the gap with an experiment that directly probes the free charge carriers.

We have carried out scanning tunneling microscopy (STM) and spectroscopy (STS) experiments on Sr_2IrO_4 . Through single-particle tunneling, STS measurements cover both occupied and unoccupied states, yielding the local density of states (LDOS) to a good approximation and hence the insulating gap directly [24, 25]. Furthermore, the spatial resolution enables us to study variation down to the atomic-scale and to search for native defects that can pin the chemical potential and affect other bulk properties. In this letter, we present the first atomically resolved topographic [26] and spectroscopic studies in Sr_2IrO_4 , uncovering a gap of 620 meV. We have also studied topography with various junction resistances, and measured the LDOS with respect to the defects and to the tip-sample separation.

In this study, single-crystals of Sr_2IrO_4 were cleaved in ultra-high vacuum (5×10^{-11} torr) immediately before being loaded into the scan-head, whose temperature is precisely stabilized at 80 K. Before the tunneling experiments, we verified that the density of states of the tips were featureless. For this data the junction was mea-

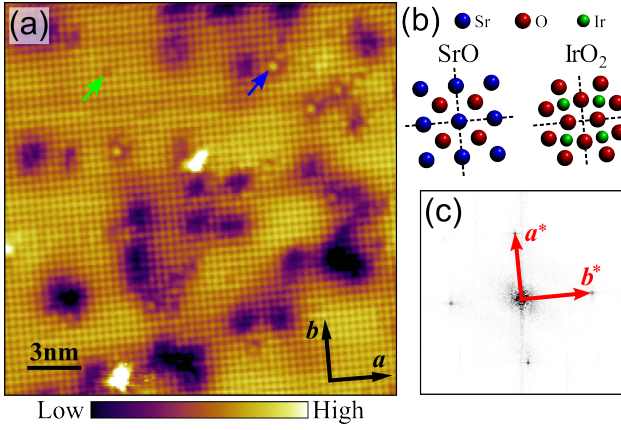


FIG. 1. (Color online) A $20 \times 20 \text{ nm}^2$ topographic image taken with -300 mV and 5 pA at 80 K (a) and its Fourier transform (c). The black arrows indicate the a and b axes. Green (gray) and blue (dark) arrows indicate two Sr-related defects. (b) Schematic diagrams of the first SrO and second IrO₂ layers. The square lattice in (a) correspond to Sr.

sured to have a work function of $3.6 \pm 0.2 \text{ eV}$, which is necessary for measurements with bias voltages up to $\pm 1 \text{ V}$. Due to the insulating nature of Sr₂IrO₄ measurements with high temperature and with large junction resistance ($> 10 \text{ G}\Omega$) are necessary (see below) to only minimally perturb the surfaces. Furthermore, Sr₂IrO₄ cleaves between two SrO layers leaving a charge-balanced surface, unlike the different surface terminations in materials like Pr_xCa_{1-x}Fe₂As₂ [27].

After cleaved, Sr₂IrO₄ shows a SrO layer on the surface. Fig. 1a shows a $20 \times 20 \text{ nm}^2$ constant-current image, in which we can easily see the Sr square lattice (Fig. 1b). The Fourier transform (Fig. 1c) of this image shows four strong atomic peaks, confirming that this high quality surface is free of reconstruction. Together with the atomic lattice, the sample also shows a small number of defects with the most evident type being the dark patches (see below).

Shown in Fig. 2a is an LDOS spectrum measured by averaging ~ 1300 spectra taken over a $2 \times 2 \text{ nm}^2$ area that did not include any apparent defects. The negative sample-bias side of the LDOS, corresponding to the occupied states, shows no distinct feature that can be related to the lower Hubbard band (LHB) of $J_{\text{eff}} = 1/2$ band down to -1 eV (Fig. 2b). This observation may seem to contrast with the double-peak structure observed in optical conductivity measurements [23]. However, these measurements in insulators are sensitive to excitons [28] and Kim *et al.* [29] have pointed out that the double-peak seen in the optical conductivity are not due to the quartet band and the LHB. Additionally, in the related compounds Na₂IrO₃ and Li₂IrO₃, the importance of excitonic effects have been shown in a recent RIXS study [30]. It is therefore likely that the LHB is overlapping

with rather than separate from the quartet band, in good agreement with calculations done by LDA+DMFT [17] and by variational cluster approximation [14]. According to Watanabe *et al.* [14], the LHB is entirely enclosed by the quartet band. This overlap implies that the strong SOC limit and single-band models are inadequate for Sr₂IrO₄ and many-body effect should be taken into account.

The insulating gap from our tunneling spectra is about 620 meV , with the valence band top at -150 meV and conduction band bottom at $+470 \text{ meV}$ (Fig. 2a). Here the energy gap is defined as the size of the energy window without coherent density of states, and the edges of the gap are determined by linearly extrapolating the bands to vertical zeros [31]. The energy of the valence band top (-150 meV) is in excellent agreement with the one measured by ARPES [9, 22]. The slow rise in the LDOS between 0.2 and 0.5 eV varies with tunneling location and tip-induced electric field and hence we believe it to be extrinsic either due to local defect states or variable tip-induced band bending. This extrinsic nature is

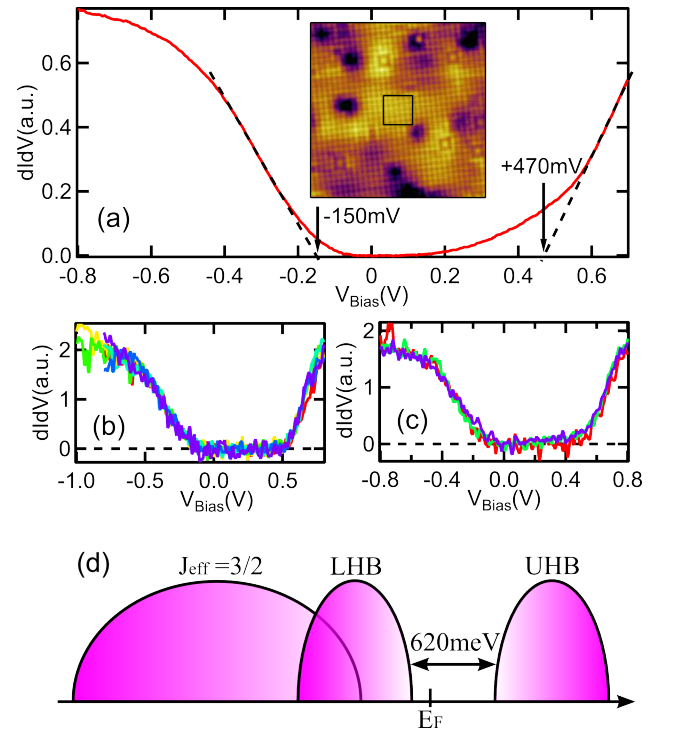


FIG. 2. (Color online) The 620 meV energy gap. (a) LDOS measured by averaging 36×36 spectra over the $2 \times 2 \text{ nm}^2$ area indicated by the square in the image (inset). The dashed lines are drawn to indicate the band edges at -150 meV and $+470 \text{ meV}$. Data was taken with -300 mV and 10 pA and ac modulation of $8 \text{ mV}_{\text{rms}}$. (b) and (c) LDOS taken at locations away from the defects, (b) without and (c) with the slow rise. (d) Diagram showing energy bands with two important features: the 620 meV insulating gap and the overlap between the LHB and the $J_{\text{eff}} = 3/2$ (quartet) band.

further confirmed by the uniformity of the 620 meV gap unlike the slowly rising LDOS inside it which is seen only in some but not all of the locations (Figs. 2b and 2c) and at different junction resistances (Fig. 3f). The reduction of this feature with decreased tip-induced electric field is consistent with it not being the intrinsic spectrum but instead due to band bending and defect related charging. We report values for the large gap that are the limits as we lower the perturbing field. Therefore, the large 620 meV gap is due to the underlying nonlocal energy bands. Furthermore, LDOS measurements in Rh-doped sample $\text{Sr}_2\text{Ir}_{1-x}\text{Rh}_x\text{O}_4$ ($x = 0.04$) [32] also confirm this by showing that far from the intentional impurities a similarly sized gap is present.

However, as discussed above, this large gap is not in good agreement with the gap values reported by other probes. It is interesting to note that in the similar material La_2CuO_4 , the debate about the gap size has lasted for a long time [33–35] because of precisely the aforementioned subtleties. The difficulty in extracting the correct energy scales of the intrinsic gap rises when other processes are involved in the experiments, as stated above. Fortunately, STS can measure the Mott gap directly through the single-particle LDOS. It does require a tunneling current through the sample and so may have difficulty for materials with poor electrical conductivity, but we have overcome this problem by performing the experiment at liquid nitrogen temperatures and with very low tunneling currents, minimizing the tips' electric field at the sample.

This 620 meV gap strongly implies that Sr_2IrO_4 is a relatively weak Mott insulator rather than a Slater insulator, since the magnetic coupling energy is merely 60–100 meV [11, 36] which is too small to account for the large gap. Additionally, even though there is magnetic ordering at below 240 K in this material, consistent with a Slater insulator, transport does not see a metallic state up to 600 K [37] showing that the magnetic ordering is not the source of the insulating nature. Although the size of this gap is considerably smaller than the 3d cuprates [25, 33], it is still large compared to what one would expect from the extended 5d orbitals and the fact that Sr_2RhO_4 , which we naively expected to have a higher U and lower W , is actually a paramagnetic fermi liquid [3, 38]. The band minimum of the upper Hubbard band (UHB) of the $J_{\text{eff}} = 1/2$ band, is the major discrepancy between our STS result and the existing studies. LDOS measurements show that the band minimum of UHB is at +470 meV, implying that LDA calculations need a larger U to capture this feature since it has been shown that the insulating gap depends on U sensitively [17]. In a recent study on Na_2IrO_3 , Comin *et al.* [39] have shown that using U as large as 3 eV in LDA calculation is necessary to reproduce the 340 meV gap in that material. With this work a stronger-than-expected correlation effect has now been observed in two iridates. The mea-

sured gap in Sr_2IrO_4 is nearly twice the measured gap seen in Na_2IrO_3 , indicating that in the material studied here the 5d electrons are even more correlated (larger U/W). Moreover, Ba_2IrO_4 , another iridate insulator [7] with a larger W due to the absence of the octahedra rotation, further confirms the strong entanglement of the energy scales in the 5d electrons.

The spatially extended nature of the 5d electrons would seem to greatly reduce the on-site Coulomb repulsion, but will concomitantly increase the same interactions between the electrons sitting on neighboring Ir sites due to the increased overlap of their wave functions. This neighboring Coulomb interaction has been theoretically studied in the extended Hubbard model [40]. For Sr_2IrO_4 , the Wannier functions calculated by Jin *et al.* [21] do show that a significant amount of electron weight is distributed on the four neighboring sites. It is thus strongly possible that the symmetric arrangement of the neighbors can result in a net repulsion that acts as an effective on-site one. Interestingly, every Ir^{4+} ion in Sr_2IrO_4 has four nearest neighbors while there are only

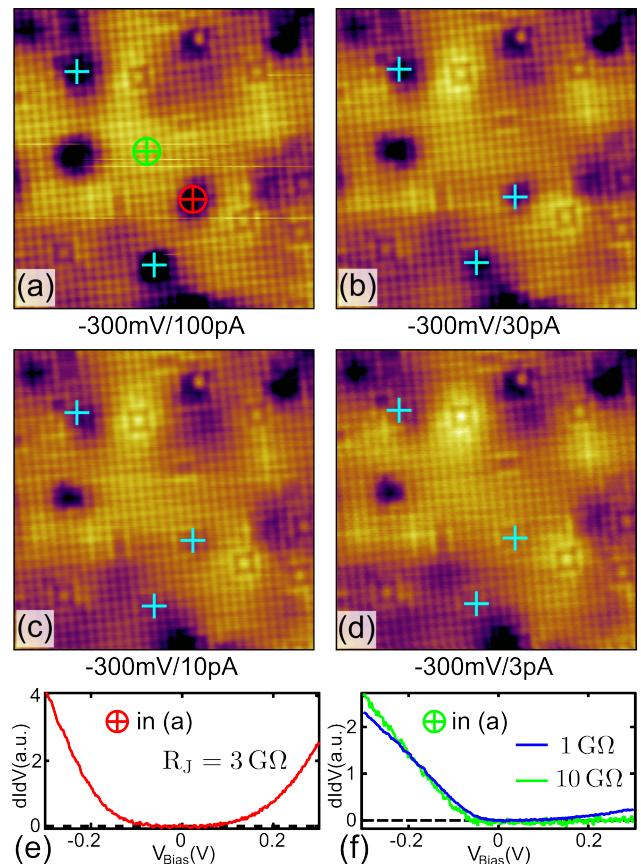


FIG. 3. (Color online) (a-d) Images with a wide range of junction resistances: 3.3–100 GΩ. The + signs indicate defects looking differently with tunneling condition. (e) LDOS measured on the red (dark) \oplus in (a). (f) LDOS measured on the green (gray) \oplus in (a) with 1/10 GΩ.

three in Na_2IrO_3 which has a smaller gap. This is in agreement with their gap sizes if the neighboring interaction is indeed causing a larger Hubbard-U.

To understand the effects caused by the defects, we have further studied topography at different junction resistances and LDOS on and off the defects. We see two types of Sr-related defects in the topography: Sr vacancy (indicated by the red arrow in Fig. 1a) and Sr ad-atom (blue arrow in Fig. 1a). These two types of defects may be caused by the cleaving process, but we find they have little effect on the local electronic structure. In stark contrast the third type of defect we see, the larger dark patches seen in the topography, changes the local electronic structure dramatically. We identify these dark-patch defects as related to oxygen defects, similar to those seen in manganites [41] and cuprates [42, 43]. Topographic images (Fig. 3) with junction resistance ranging from 3.3 to 100 $\text{G}\Omega$ confirm that the patchiness associated with these defects are due to electronic inhomogeneity rather than surface structural corrugation. The cross signs in Fig. 3a-d indicate these oxygen defect related areas that vary with different tip-sample separation. Such a high sensitivity in topography with respect to the tunneling junction reflects the poor screening in the presence of perturbations due to the insulating nature of Sr_2IrO_4 , and also the potential influence of the defects in these materials. While it is true that for some transition metal oxides, oxygen atoms in surface layers are volatile, but we didn't observe any degradation of surface quality after several days at 80 K implying the stability of the surface and these defects. Therefore, these (O related) defects are likely native to the sample, in accord with the O deficiency found in this material [44, 45], and also in $\text{Sr}_3\text{Ir}_2\text{O}_7$ and BaIrO_3 [46]. This result naturally explains why transport measurements give a smaller gap [19, 20] and optical conductance shows a slow rise starting at 0.2 eV [23]. The presence of these defects leads to in-gap states, lowers the effective gap measured by transport and gives a lower energy scale for optical transitions. More importantly, the random distribution of the defects is in good agreement with the variable-range-hopping behavior in electrical conductivity [6]. The existence of native defects despite the good sample quality and our ability to measure the intrinsic LDOS apart from the influence of defects, prove the necessity of using a local probe like STM to study this material.

Lastly, the LDOS study on top of a defect (Fig. 3e) shows how strongly the tip's electric field can change the density of states around E_F by building up in-gap states. It should be pointed out that, due to the dramatic difference in topography of this defect as we mentioned earlier, the STS measurement may not be simply reflecting the underlying LDOS, but it does serve as evidence for the in-gap states around them. Fig. 3f shows the subtle variation in the measured spectra related to the tip-induced band bending, reinforcing the necessity of measuring with

high junction resistances to reduce the perturbing electric field of the tip.

In summary, we have presented the first atomic-scale study in Sr_2IrO_4 with density of states measurement covering the entire Mott gap, which is 620 meV. The measurement of the occupied states has confirmed the overlap between LHB and quartet band. We conclude that Sr_2IrO_4 is a Mott but not Slater insulator by comparing the gap size with the magnetic coupling energy, implying the stronger-than-expected el-el correlation in this 5d system. Furthermore, we suggest that additional source of Coulomb repulsion needs to be taken into account to qualitatively and quantitatively understand the 5d electron systems. Lastly, the junction dependence of the defects in the topography, combined with the LDOS results around them, indicate their profound impacts on the local electronic structure of this insulator. Our results strongly imply that in Sr_2IrO_4 the three energy scales (SOC, U and W) are comparable to each other and that studies of the 5d electrons should put all of them on similar-footing.

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Note added – After this manuscript has been prepared, we became aware of a similar work by Nichols *et al.* [47].

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